

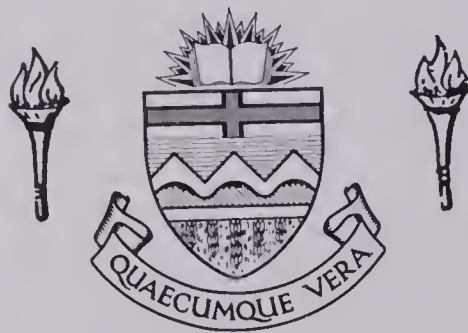
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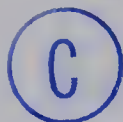
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UNIVERSITY OF ALBERTA

THE EFFECT OF SULFUR CONCENTRATION
ON
FRICTION AND WEAR OF IRON-SULFUR ALLOYS

by



Pradip Kumar Roy

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES
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The undersigned certify that they have read, and recommend to
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THE EFFECT OF SULFUR CONCENTRATION
ON
FRICTION AND WEAR OF IRON-SULFUR ALLOYS

Submitted by

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in partial fulfilment of the requirements for the degree of Master of
Science.

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ABSTRACT

Friction and wear characteristics of iron containing varying amounts of sulfur from 0 to 8.14 weight percent were studied under normal atmospheric conditions. Friction and wear studies were conducted with a 0.3 inch diameter cylindrical rider sliding in a circular path on different plates at various speeds and loads. Additions of small amounts of sulfur to pure iron reduced friction and wear considerably and at higher sulfur concentrations there was no appreciable reduction in friction and wear. The friction coefficient increased slightly with increasing normal load. Increased sliding speed reduced the friction coefficient and the wear rate. Friction and wear of iron-sulfur alloys were increased with rising hardness of the plate materials. The bulk hardness and the friction coefficient were expressed by power laws in terms of the sulfur content of the alloy.

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INTRODUCTION

With the recent advances in the field of electronics, instrumentation and aerospace in particular, the lubrication of various components under severe environmental conditions like nuclear radiation, ultra high vacuum, extreme temperature and pressure has become important. Sliding surfaces are being subjected currently to an increasing range of thermal, mechanical and environmental stress conditions, posing new problems in friction and wear. Conventional mineral oils and even the recent synthetic fluid lubricants can not work effectively above 350°C due to their high evaporation rates, further, at low temperatures hydrocarbon lubricants become often contaminated, making them unsuitable for certain applications.

The work of Lancaster (1966) and Campbell, Loser and Sneegas (1966) shows the advantages of using solid lubricants for many applications rather than fluid lubricants. There are two methods of employing solid lubricants- the first one, is the lubrication of sliding surfaces by interdeposition of a film of solid lubricant, the other, which is the better one, employs composite materials containing the solid lubricant. Both methods can reduce friction and wear under severe environmental conditions by virtue of their good chemical, mechanical and dynamic stability.

The importance of low shear strength, work hardening and flexural fatigue characteristics of solid lubricants for application in space were stated by Lewis (1963), where problems of extremely low pressure; zero gravity, sub gravity or over gravity; nuclear and cosmic radiation; particle impact in various planetary gaseous environments are encountered. Although the use of graphite as a lubricant probably dates back to the

middle ages, the use of bonded solid lubricant materials is relatively new. The use of MoS_2 as a lubricant started in the 1940's. It is currently the most popular solid lubricant and extensive research by Saloman, DeGee and Zaat (1964), Haltner (1964), Karpe (1965) and Lancaster (1967) has shown that it can reduce friction considerably by virtue of its lattice layered structure.

Sikorski (1964), Buckley and Johnson (1966) and Rabinowicz (1967) in their recent work correlated friction, wear and adhesion with crystal structure, surface energy-penetration hardness ratio, atomic size, shear strength and slip behavior.

Iron sulfide has a NiAs type of structure with iron atoms in $001; 001/2$ and sulfur atoms in $1/3 \ 2/3 \ 1/4; \ 2/3 \ 1/3 \ 3/4$ positions (Wyckoff, 1963), with lattice constants of 3.438 Å and 5.880 Å and a "c/a" ratio of 1.71. Its hardness is about 219 KHN and that of pure iron is about 75 KHN. Iron sulfide was chosen as a solid lubricant because of its high hardness and "c/a" ratio. High "c/a" ratios lead to low friction coefficients (Buckley and Johnson, 1966). High hardness appears to be desirable because the most frequently used solid lubricants, MoS_2 and graphite, have hardnesses in excess of 1000 KHN (Lancaster, 1966).

Usually iron sulfide is obtained by surface treatments (Sakuri, Ikeda and Okabe, 1965). However, alloying techniques can also be employed.

Buckley and Johnson (1963, 1964) using this alloying technique studied friction, wear and welding characteristics of iron-sulfur alloys and some sulfur modified steels at pressures from 760 to 10^{-9} mm.Hg. They investigated those characteristics with a hemispherical rider, which slides in a circumferential path on the flat surface of a rotating metal disc of the

same material, at a constant normal load of 1000g. and sliding speeds from 28 to 1000 fpm. They observed that the presence of relatively small quantities of iron sulfide reduces friction and wear of pure iron considerably.

The object of the present thesis is to study in more detail the effect of sulfur concentrations on friction and wear properties of pure iron under atmospheric conditions with different contacting surfaces, sliding speeds and normal loads.

GENERAL REVIEW

A. HISTORICAL DEVELOPMENT

The phenomenon of friction is important, as it represents a universal attribute of matter.

The first application of friction, namely the use of frictional heat, has its roots in prehistory. The second application showing understanding of frictional phenomena, namely the use of sleds, rollers or wheels, often supplemented by liquid lubricants to minimize the work required to transport heavy objects, dates back more than 3000 years.

The scientific study of friction is, however, much more recent than these applications might suggest.

Early investigators of friction include Amontons (1699), Coulomb (1785) and Morin (1833). They hypothesized that friction is due to the intermeshing of mechanical protuberances or asperities on the surfaces of the contacting materials. They derived the classical laws on the basis of the "roughness hypothesis", summarized below:

1. The frictional force is directly proportional to normal load.
2. The frictional force is independent of the contact area and the sliding velocity.
3. The frictional force depends upon the nature of the contacting materials.

The "roughness hypothesis" remained the majority view right through the nineteenth century. Beginning about 1920, however, interest began to revive the "adhesion hypothesis" after the work by Hardy (1919) and Tomlinson (1929). By this time the science of surface chemistry was well developed to examine the frictional properties of surfaces with different degrees of contamination. The large differences of friction produced by

varying the contamination was easily explained in terms of the "adhesion hypothesis".

The "adhesion hypothesis", however, could not explain the dependence of the frictional force on the area of contact, until the conception of "real area of contact" was introduced by Ernst and Merchant (1940) and Bowden and Tabor (1942). The real area of contact between two contacting bodies is made up of a large number of discrete regions of contact. Almost at the same time another school of workers proposed the electrostatic theory of friction (Schnurmann, 1942). When contacts are broken and reformed during sliding triboelectric forces come into play causing frictional phenomena. This theory was able to explain the phenomenon of stick slip and the decrease of friction at high sliding speed, which the classical school could not explain. In spite of extensive studies in friction, there is hardly any unique theory of friction, however, the work of McFarlane and Tabor (1950); Rabinowicz and Tabor (1951); Tylecote, Howd and Furmidge (1958); Anderson (1960); Rabinowicz (1965); Kragelski (1965) and Rabinowicz (1967) has resulted in general acceptance of the adhesion theory.

The history of wear may be presented in very short form, for although the phenomena must have been first observed many years ago, their systematic study is far more recent, in spite of the fact that it is a problem of greater technological importance. Systematic studies on wear started in the late 1940's, after the discovery of radio isotopes, which make the study of wear possible while it is happening. Important contributions to the laws of adhesive wear were made by Rabinowicz and Tabor (1951); Rabinowicz and Shooter (1952); Rabinowicz (1953); Archard

(1953); Greenwood and Tabor (1955) and Rabinowicz (1964). Kragelski (1965), however, described the wear to be essentially a fatigue process.

B. FRICTION

The Origin of the Friction Force

Contacts between two surfaces are usually discrete and the interaction between the surfaces at these spots is of a dual molecular-mechanical nature (Kragelski, 1965). The irregular surface contour enables some points to approach each other causing mutual repulsion, while others remain further apart and attract each other due to the action of forces of molecular interaction. These forces are caused by the electromagnetic field existing inside the body which gives rise to standing and travelling waves that interact with the opposing surface. These forces form very strong adhesive bonds (or frictional bonds) at the points of real contact. When sliding occurs these frictional bonds get destroyed and reform again. The mechanical interaction is due to the intermeshing of gross surface asperities which cause the bulk deformation of the solids owing to the change in the state of stress. When the strength of the adhesive bond is less than that of the underlying layers a "positive gradient" of mechanical properties is said to exist- which is a necessary condition for external friction.

The Magnitude of the Friction Force

Bowden and Tabor (1964) have shown that when two sliding solid surfaces are pressed together by a load "L" normal to their general interface, the sliding will be induced by a frictional force "F". They

assumed an average shear stress " s_{av} " and yield stress " p " over the real area of contact and showed -

$$f = s/p \text{ ----- Eq. (1)}$$

Since, " s_{av} " can not exceed substantially the bulk shear strength " s " of the softer of the two contacting materials.

The value of the friction coefficient in Eq. 1 depends on the yield criterion used to relate the shear stress to the yield stress.

Friction is also affected by contaminants at the interface, so that experiments carried out in air poorly agree with Eq. (1), because the results depend on the properties of the contaminants rather than the substrate. But the agreement becomes progressively better when the contaminants are removed by carrying out tests in dry air, then in vacuum, then, after outgassing at elevated temperatures, in a high vacuum. Tests carried out with clean surfaces, even in high vacuum, do not agree with Eq. (1) and the friction coefficient varies with the surface geometry (Bowden and Hughes, 1939; Gwathmey, 1951 and Bowden and Young, 1951). Further, in obtaining Eq. (1), surface energy effects have been ignored. The real area of contact tends to be larger with the increase in surface energy of the two surfaces in contact. This effect is pronounced if the energy of adhesion " W_{ab} " between two surfaces is high and the penetration hardness of the softer material " p " is low. The increase in real contact area leads to an increase in the frictional force required to shear the junction. A generalized expression for the friction coefficient (Rabinowicz, 1967) using the surface energy criterion is,

$$f = s/p + c.W_{ab}/p \text{ ----- Eq. (2)}$$

where, "c" is a constant for the same surface roughness, applied load and sliding speed, and " W_{ab} " depends both on the sliding materials and their interaction.

C. WEAR

Mechanism

In normal conditions of sliding, it is essential to ensure a positive gradient of mechanical properties beneath the surface which is only possible when surface films are formed to protect the substrate material from direct contact. In unlubricated conditions, the oxide film continuously forms, grows to a critical thickness, peels off and reforms. The films protect the substrate material but do not prevent subsurface deformation as a consequence of interpenetration of the surface asperities. Each asperity is associated with a wave of deformation; the material surface immediately in front of the asperity is compressed and the material surface behind the asperity is elongated due to the frictional force. Thus, each cross-section of the sliding member is successively subjected to compressive and tensile stresses (elastic or plastic), which cause subsurface cracks.

In general, wear is made up of two parts: that due to fatigue of the substrate material and that due to removal of the oxide film, which is also essentially a fatigue process.

In lubricated conditions, a fatigue failure may also occur because the lubricant does not remove the load acting on the surface but merely

balances it. In addition, a lower value of the frictional force reduces the tensile stress, and therefore also decreases wear by fatigue (Kragelski, 1965).

Laws of adhesive wear

On the basis of the work of Rabinowicz and Tabor (1951); Archard (1953) and Greenwood and Tabor (1955) certain quantitative laws of adhesive wear were established.

1. The amount of wear is generally directly proportional to the load " L ".
2. The amount of wear is generally proportional to the distance slid " x ".
3. The amount of wear is generally inversely proportional to the hardness " p " of the surface being worn away.

$$V = c.L.x/p \text{ -----Eq. (3)}$$

Where, " V " is the volume of the material worn away and " c " is a non-dimensional constant dependent on the materials in contact and their exact degree of cleanliness.

Rabinowicz (1964) using the surface energy criterion evaluated the size of loose wear particles. He assumed a model in which an asperity with a flat end slides over another surface, adheres at one spot and then shear occurs within the asperity so that a hemispherical loose particle is formed. The wear particle carries high compressive stress so long as it is attached to the asperity. The normal stress does not act after the fragment formation but the horizontal strains remain locked - in due to residual frictional stresses at the interface. Because of the locked - in strains, the particle is associated with an elastic volume energy " E_v " and

a surface energy " E_s " locked up at the interface. For a wear particle to become loose, " E_v " has to be greater than " E_s ". Applying this condition,

$$d = 6.E.W_{ab}/v^2.s_y^2 \text{ ----- Eq. (4)}$$

where,

E = Young's modulus.

v = Poisson's ratio.

W_{ab} = Energy of adhesion of the two sliding surfaces.

s_y = Yield stress.

d = Minimum diameter of the wear particle.

D. SOLID LUBRICATION

Devine, Cerini and Stallings (1967) define "solid lubrication" as means for the reduction of friction and wear between surfaces in rolling and sliding contact without employing hydrodynamic effects. The following factors are very important in selecting solid lubricant films -

1. Thickness: For soft films, the thickness should be as low as possible in order to prevent excessive plastic flow which may destroy the geometry of the bearing.
2. Shear strength: Should be as low as possible.
3. Adhesive and cohesive characteristics: Should be favorable in order to maintain a low rate of wear.
4. Work hardening and flexural characteristics: Should be very low, as excessive work hardening result in rapid fatigue of the film.
5. Thermal properties: Should have high conductivity, as one of the primary functions of a lubricant is the capability of dissipating the

heat generated during sliding at a fast rate. Superimposed on these lubricant film properties the actual operating parameters like load, speed, bearing clearance, temperature and pressure must also be considered.

Mechanism of boundary lubrication

According to Bowden, Gregory and Tabor (1945), the overall friction coefficient (f) of two metal surfaces lubricated by a coherent surface film of thickness comparable with the size of the asperities, is a linear function of the friction coefficients for the fully lubricated (f_1) and the unlubricated (f_m) sliding condition.

$$f = a.f_m + (1-a).f_1$$

Where, "a" is the fraction of the load supported by the substrate.

Rabinowicz (1965) proposed two models in boundary lubrication which obey this linear relationship:

(1) The single penetration model: This case occurs when the lubricant layer is penetrated by the asperities at one point only.

(2) The multiple penetration model: When the lubricant layer is penetrated at many points of the real area of contact by the asperities.

EXPERIMENTAL

A. ALLOY PREPARATION

Specimens of approximately 10 g., containing nominally 0,0.2,1.0, 1.5,2.5,4.0,6.0 and 8.5 wt. pct. sulfur flower in pure iron powder were prepared. Appendix I lists the form, purity and supplier of these materials. Silica crucibles of 0.4 in. inside diameter and 1.4 in. height were prepared from Vycor tubing.

Powdered specimens were rammed into the crucible until approximately 0.8 in. of the crucible was filled with the powder. The composite was then melted in the water cooled copper levitation coil of a Philips High Frequency Induction Furnace. The molten alloys were thoroughly mixed by the eddy current for 30 secs. and immediately cooled in air to minimize oxygen and nitrogen pick up. The cast alloys were machined to 0.3 in. by 0.3 in. cylinders. In one of the ends a cylindrical hole was drilled of 1/8 in. height and 1/8 in. diameter to accommodate the rider of the Friction Machine. The other end of the specimen constituted the rubbing surface. Ten specimens for each composition were prepared and analysed both by chemical and X-ray fluorescence techniques.

The results of the analyses were as follows:

Nominal wt. pct.	Analyzed wt. pct.
0.0	0.000
0.2	0.180
1.0	0.904
1.5	1.130
2.5	2.005
4.0	3.550
6.0	5.870
8.5	8.140

The flat surfaces of the specimen were ground on silicon carbide grinding paper of decreasing coarseness down to no. 600 grit size. They were then thoroughly washed in acetone immediately before the test. Chemical analyses, hardness measurements and metallographic observations showed very little variation for the specimens coming from the same composition lot, since the casting procedure was identical for all specimens.

B. APPARATUS

The apparatus consisted of three units:

- a. Power supply for the bridge.
- b. Friction apparatus.
- c. Recording apparatus.

a. Power supply for the bridge

1. 12-V Lead Battery: To supply the bridge excitation voltage.
2. Variable resistor: To keep bridge voltage and current constant at 4V and 30mA.
3. D.C. ammeter: Connected in series with the battery via variable resistor as shown in fig. 2.

b. Friction apparatus: Consisted of the following components -

1. Instrumented Lever Arm

Made from AISI-W2 drill rod steel, heat treated to a hardness of Rockwell C-55. The dimensions of the arm were 10 1/4 in. X 3/16 in. X 3/16 in. and it contained eleven 3/32 in. diameter holes spaced equally at intervals of 1/4 inch. These holes permitted to position the specimen holder anywhere above the disc. The specimen holder had a hemispherical tip 1/8 inch in diameter to hold the specimen and was attached to the arm by two lock nuts, which were used for fine adjustment of the normal load. A set of four Budd-foil strain gauges were attached to the sides of the arm. They formed the two active arms of the Wheatstone Bridge, shown in fig. 2 and were used to measure the normal and frictional force.

2. Post for the lever arm served as the fixture for the lever arm, the dummy gauges and the coarse adjustment of the normal load.

3. Plate materials

Glass-plate: Polished Pyrex glass-plate 1/4 inch thick and 8 inches in diameter.

Low-carbon steel-plate: Normalized to Rockwell B-65 hardness, ground with 320 grit silicon carbide paper, 1/4 inch thick and 8 inches in diameter.

Stainless-steel-plate: Annealed AISI-304 type with a hardness value of Rockwell B 82, ground with 320 grit silicon carbide paper, 1/8 inch thick and 8 inches in diameter.

Hardened-4340-steel-plate: Hardened-4340-AISI-steel with a hardness value of Rockwell C-48, ground with 320 grit silicon carbide paper, 1/4 inch thick and 8 inches in diameter.

These plates were attached to the drive shaft.

4. Drive: A 1/20 h.p. Synchronous Motor for 30 fpm. sliding speed and a A.C.-D.C. Motor for 240 fpm. sliding speed.

C. Recording Apparatus: EUW-20-A Heath Serve Recorder.

C. PROCEDURE

The output of the strain gauge bridges was calibrated with weights of 200 to 1300 gms. using a bridge excitation of 4V and 30mA.

1. The specimen was held in position with the grinding marks normal to the direction of plate rotation by the specimen holder and the normal load was adjusted by operating both the coarse and fine adjustment screws. All measurements were taken at intervals of five minutes in order to obtain a representative average value of the friction coefficient. Both the specimen and plate were thoroughly washed with acetone whenever the specimen, load, speed and the plate material were changed.

2. The wear rate was determined by measuring the weight loss of the specimen in 1/2 hour.

3. Hardness measurements for all the specimens were taken on a Rockwell Hardness Tester using the "F" scale of the machine.

4. X-ray diffraction was used to identify the phases present in the microstructures.

RESULTS

The experimental results had an estimated accuracy of about 3%. They are presented in figures 3 to 12 and in tables 1 to 12 of the Appendix-2.

The dependence of the friction coefficient on the sulfur content of the alloys at various normal loads from 50 to 850 g. and sliding speeds of 30 and 240 fpm. is shown in figures 3 to 6 and tables 1,2,3,4,5,6,7 and 8.

The dependence of the wear rate ($\text{g./cm}^2/\text{cm}$) on the sulfur content of the alloys at a constant normal load of 400 g. and sliding speeds of 30 and 240 fpm. is shown in figures 7 and 8 and tables 9 and 10.

Figures 9 and 10 show the variation of the friction coefficient with the normal load for the alloy with 8.14 pct. sulfur at sliding speeds of 30 and 240 fpm. respectively for different plate materials. Fig. 11 shows the dependence of the Rockwell-F hardness on the sulfur content in the alloys.

Figures 13 to 16 show the microstructures of a few representative specimens in the polished condition at a magnification of 450X.

At low sulfur concentrations alpha iron is present in the form of equiaxed crystals containing the FeS phase as discrete particles. At sulfur concentrations in excess of 1 to 2 percent alpha iron forms a dendritic matrix which is surrounded by a brittle network of FeS. In addition, minute quantities of Fe_2O_3 are present in the alloys form a duplex structure with FeS in the grain-boundaries. X-ray diffraction studies of alloy powder revealed only the presence of the major constituents, namely, iron sulfide and alpha-iron. The Fe_2O_3 phase, however, was detected by X-ray diffraction after its chemical extraction by Raybeck and Pasztor's (1966) technique using bromine-methyl acetate solvent.

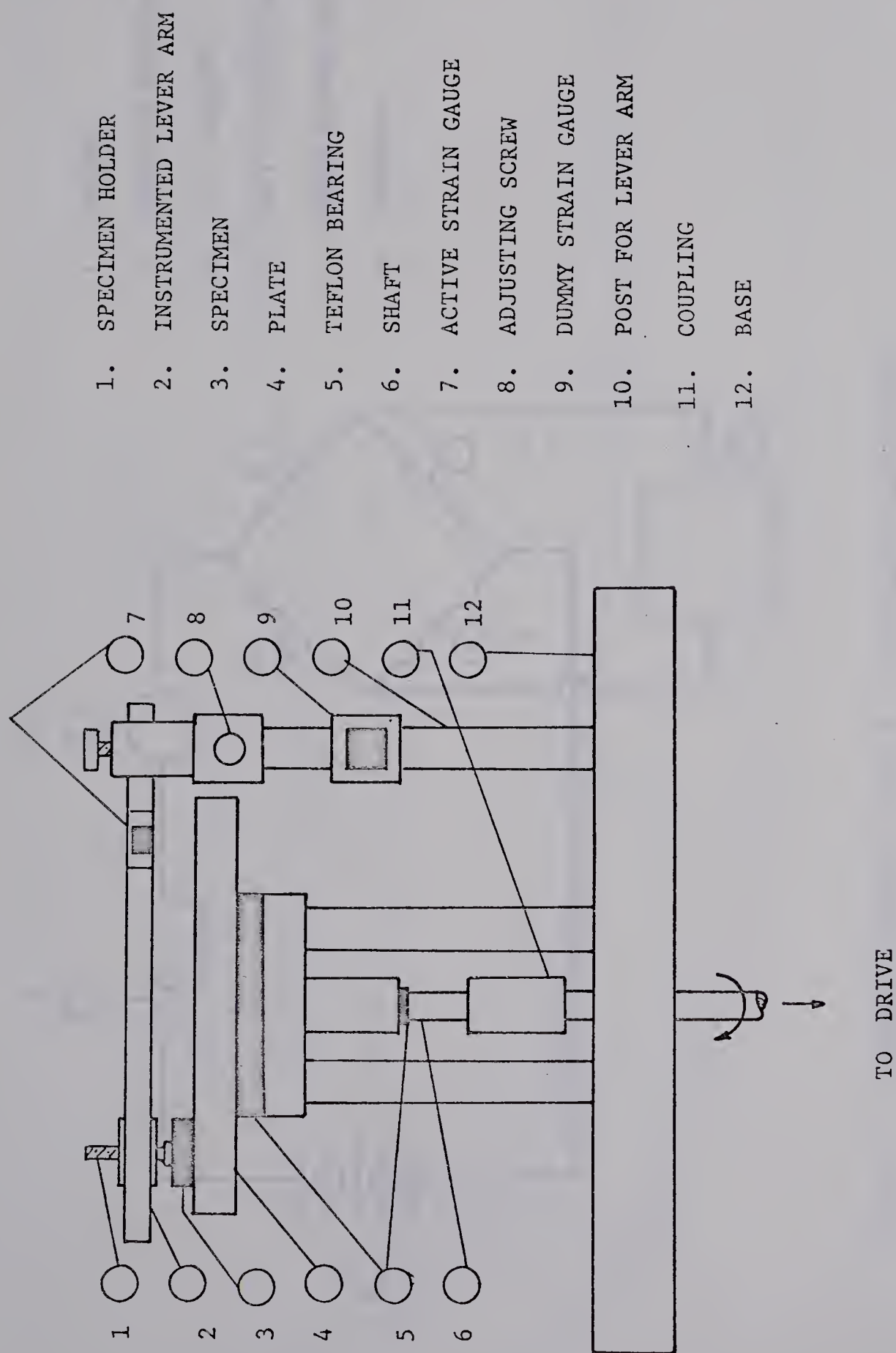


FIG. 1 - SCHEMATIC DIAGRAM OF THE FRICTION APPARATUS.

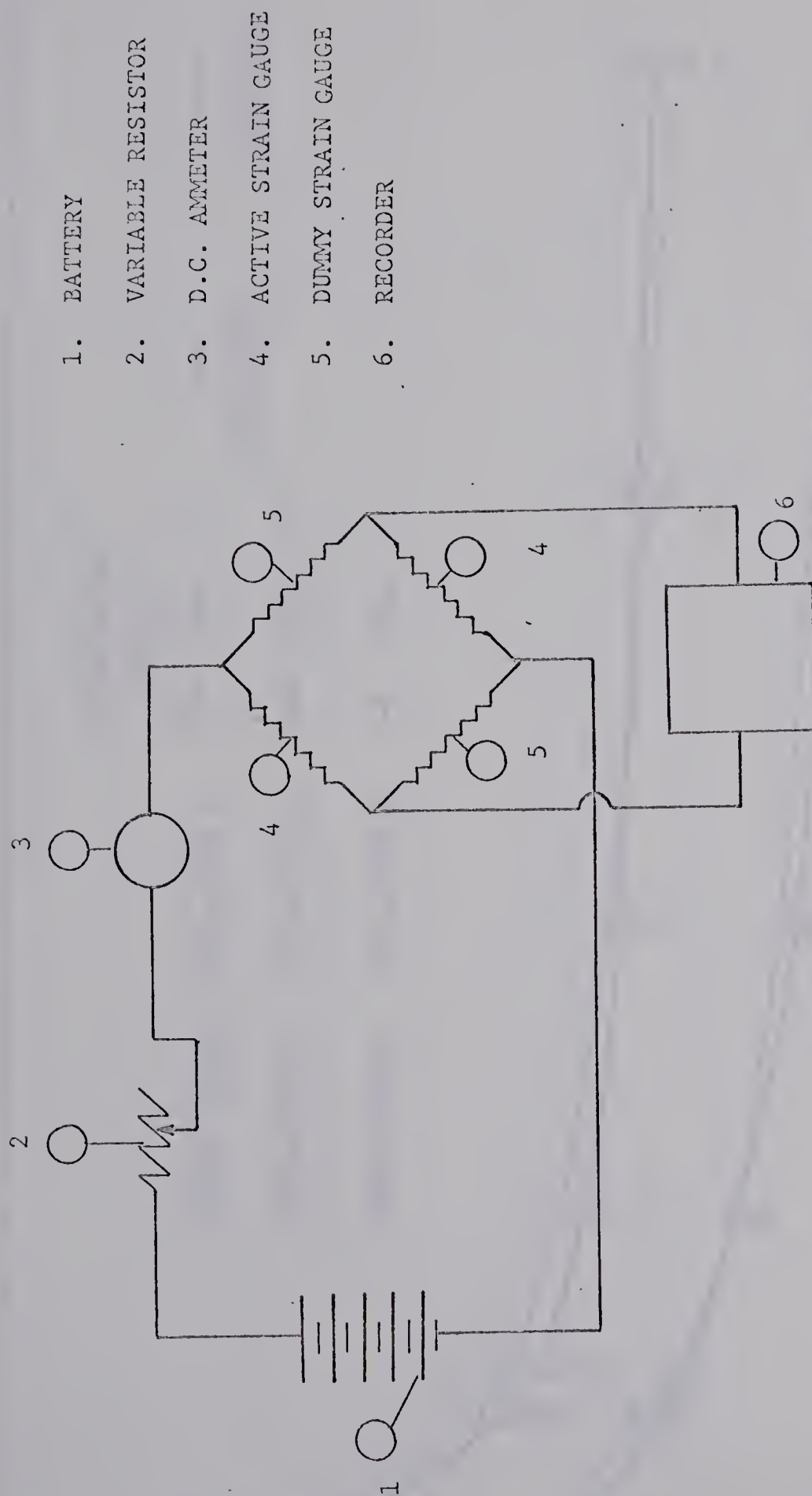


FIG. 2 - CIRCUIT DIAGRAM OF THE 4 - ARM STRAIN GAUGE BRIDGE

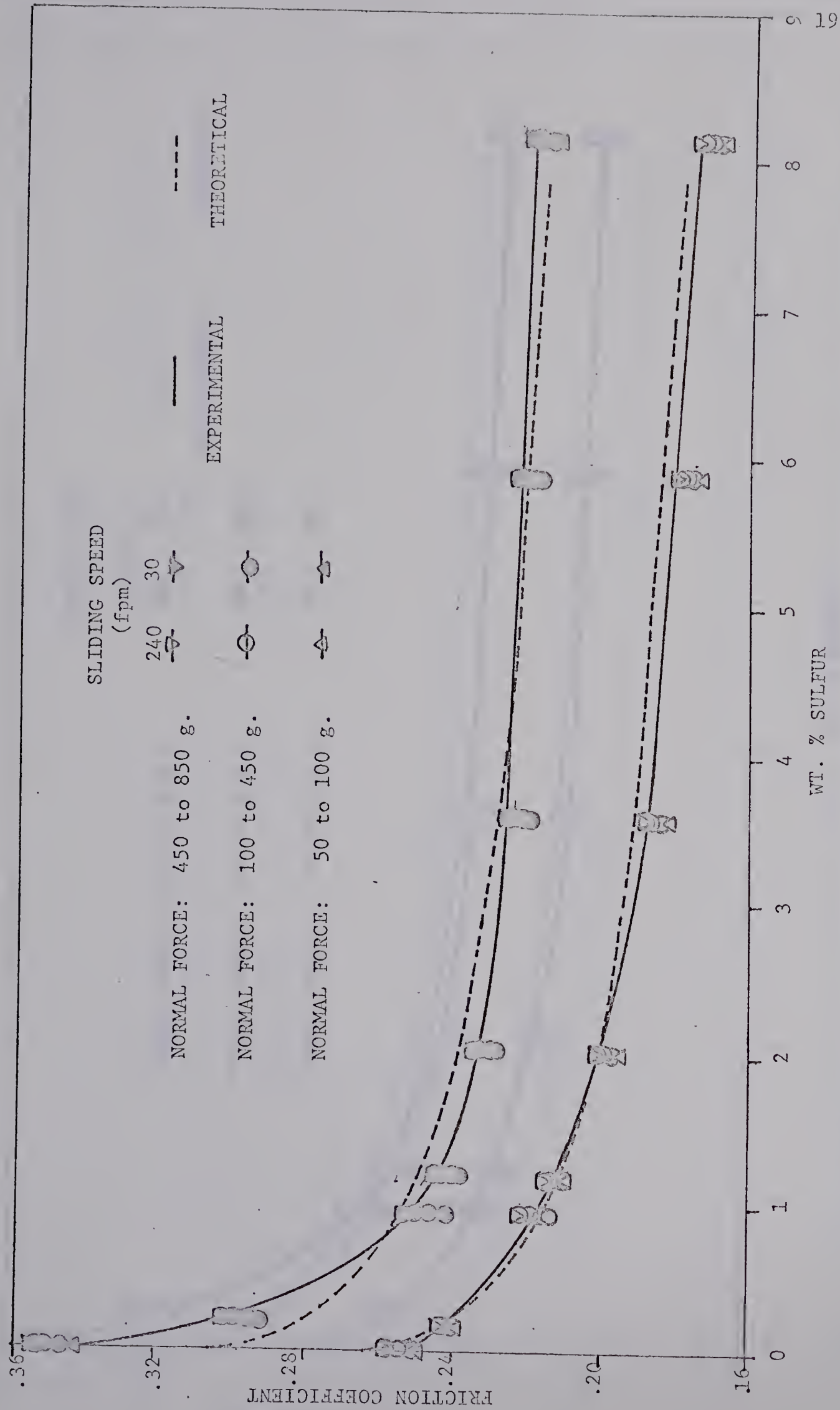


FIG. 3 - EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE ON FRICTION COEFFICIENT FOR SPECIMEN ON GLASS PLATE

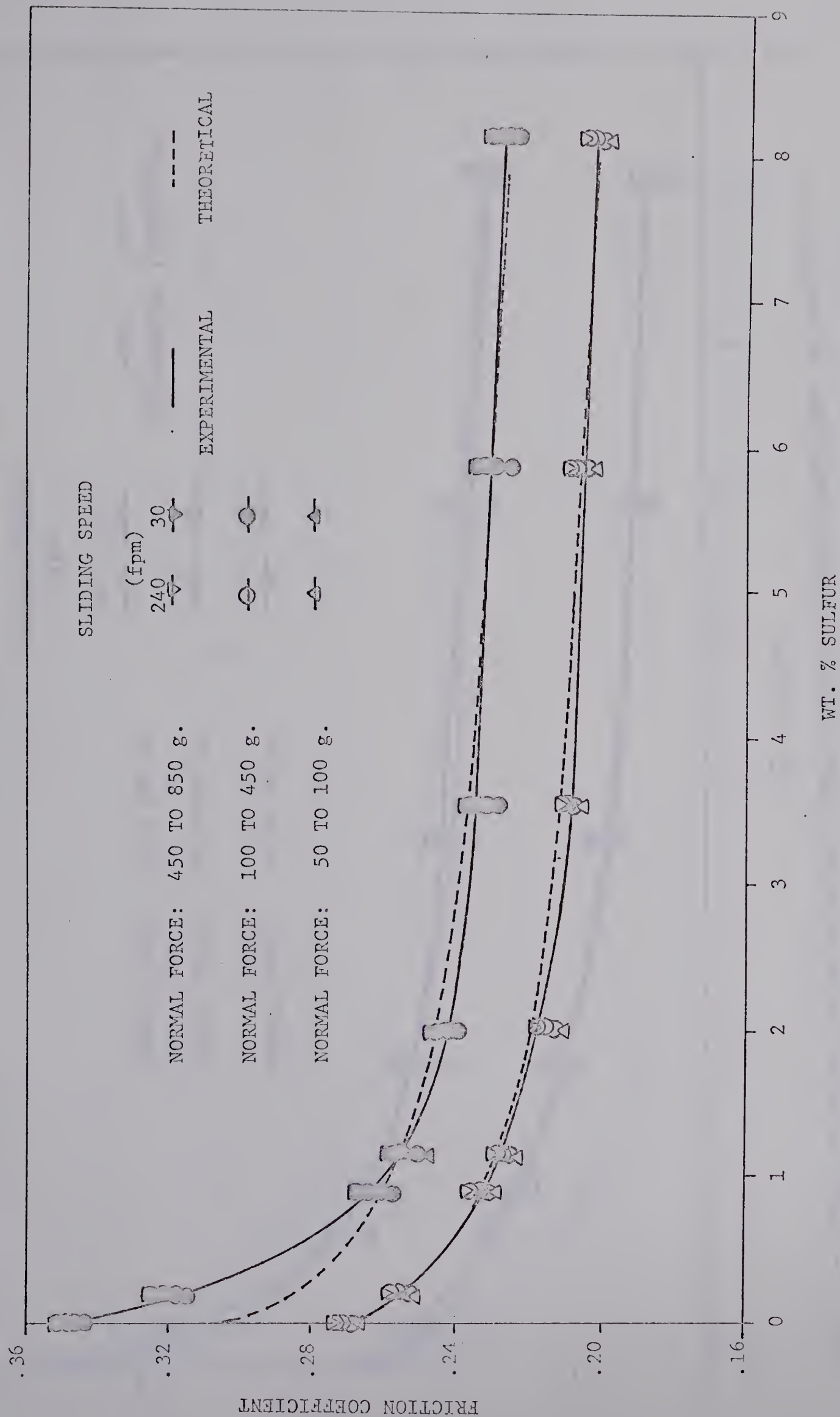


FIG. 4 - EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE ON FRICTION COEFFICIENT FOR SPECIMEN ON LOW CARBON STEEL PLATE

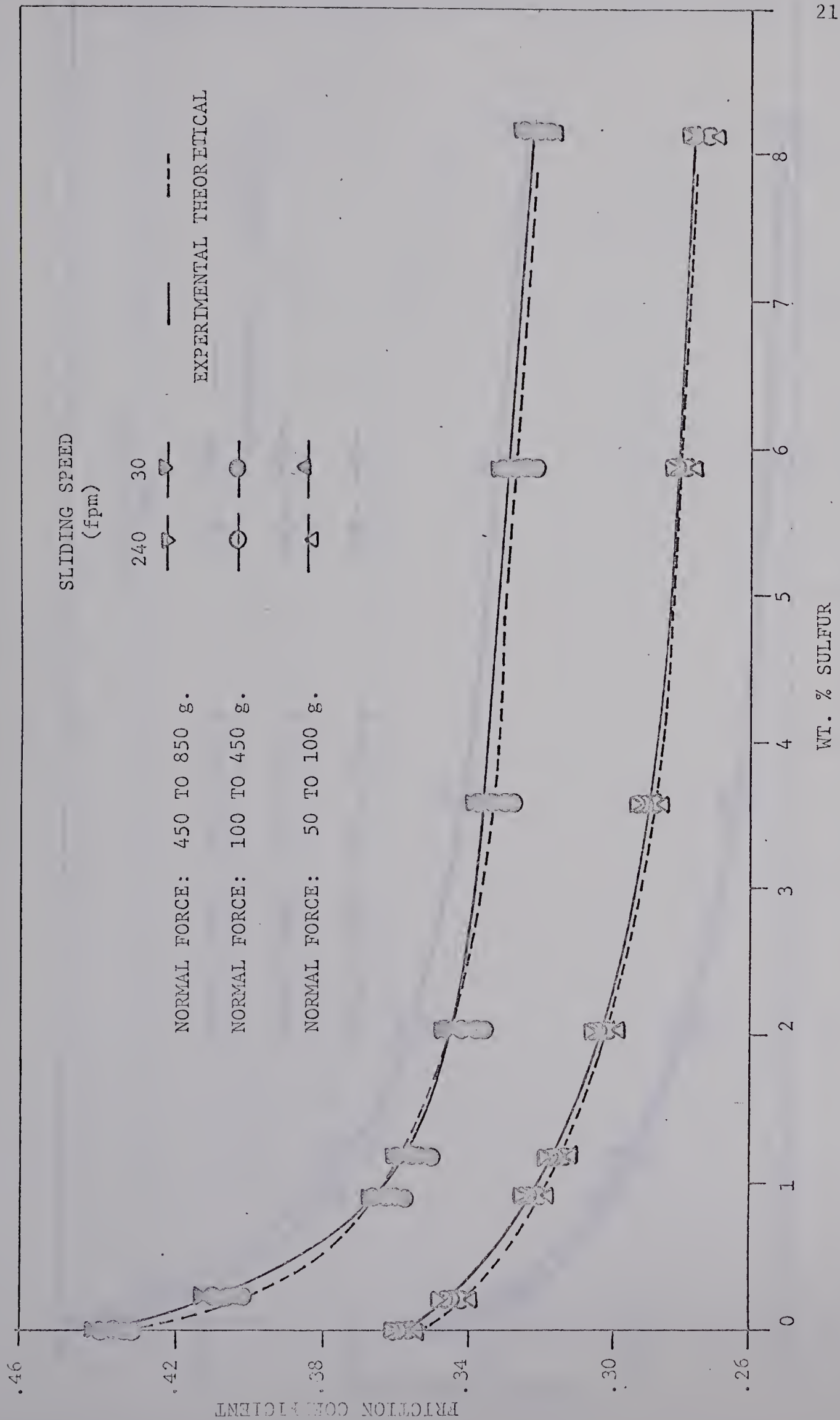


FIG. 5 - EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE ON FRICTION COEFFICIENT FOR SPECIMEN ON 304-STAINLESS STEEL PLATE

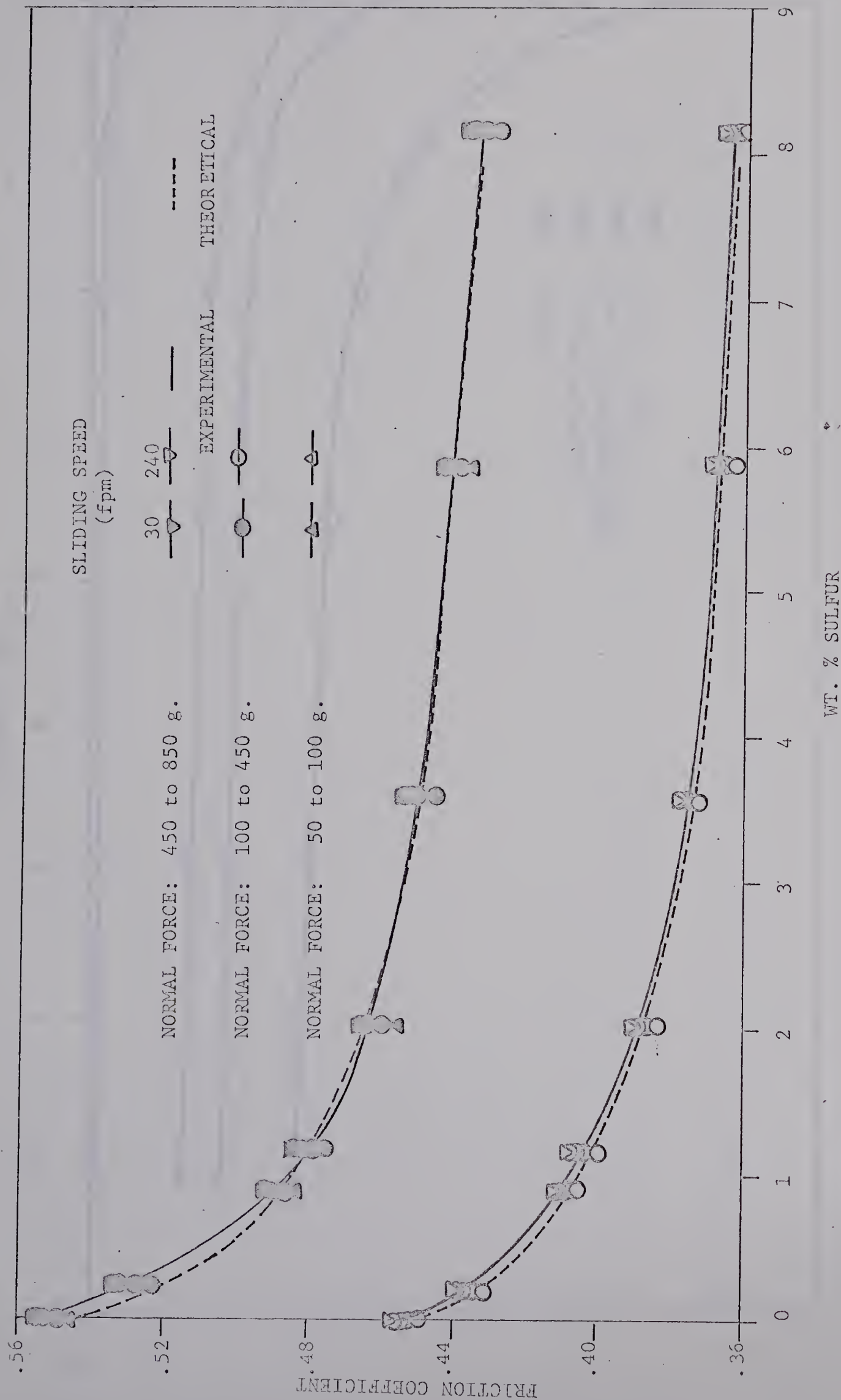


FIG. 6 - EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE ON FRICTION COEFFICIENT FOR SPECIMEN ON HARDENED 4340 STEEL PLATE

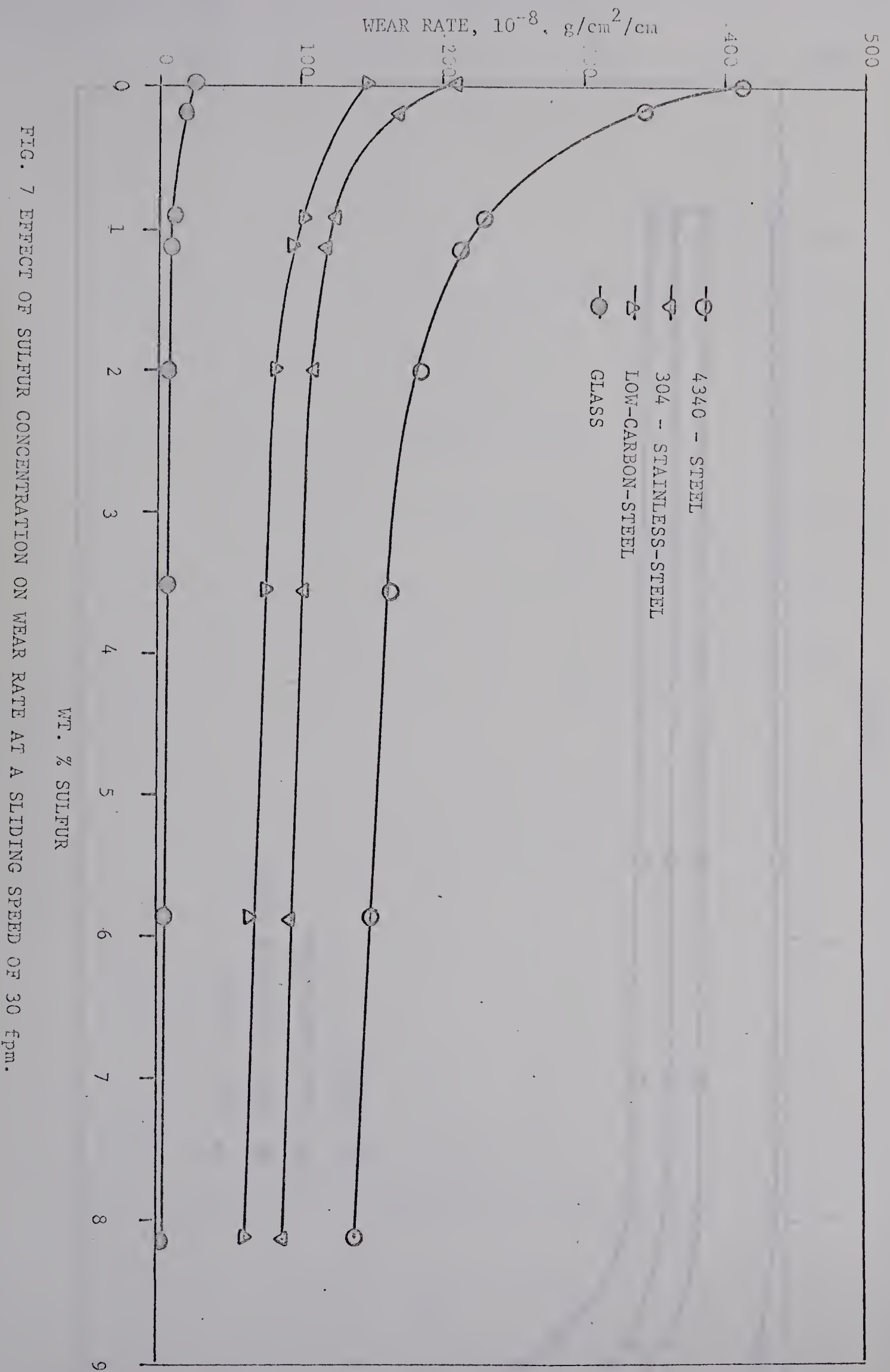


FIG. 7 EFFECT OF SULFUR CONCENTRATION ON WEAR RATE AT A SLIDING SPEED OF 30 fpm.

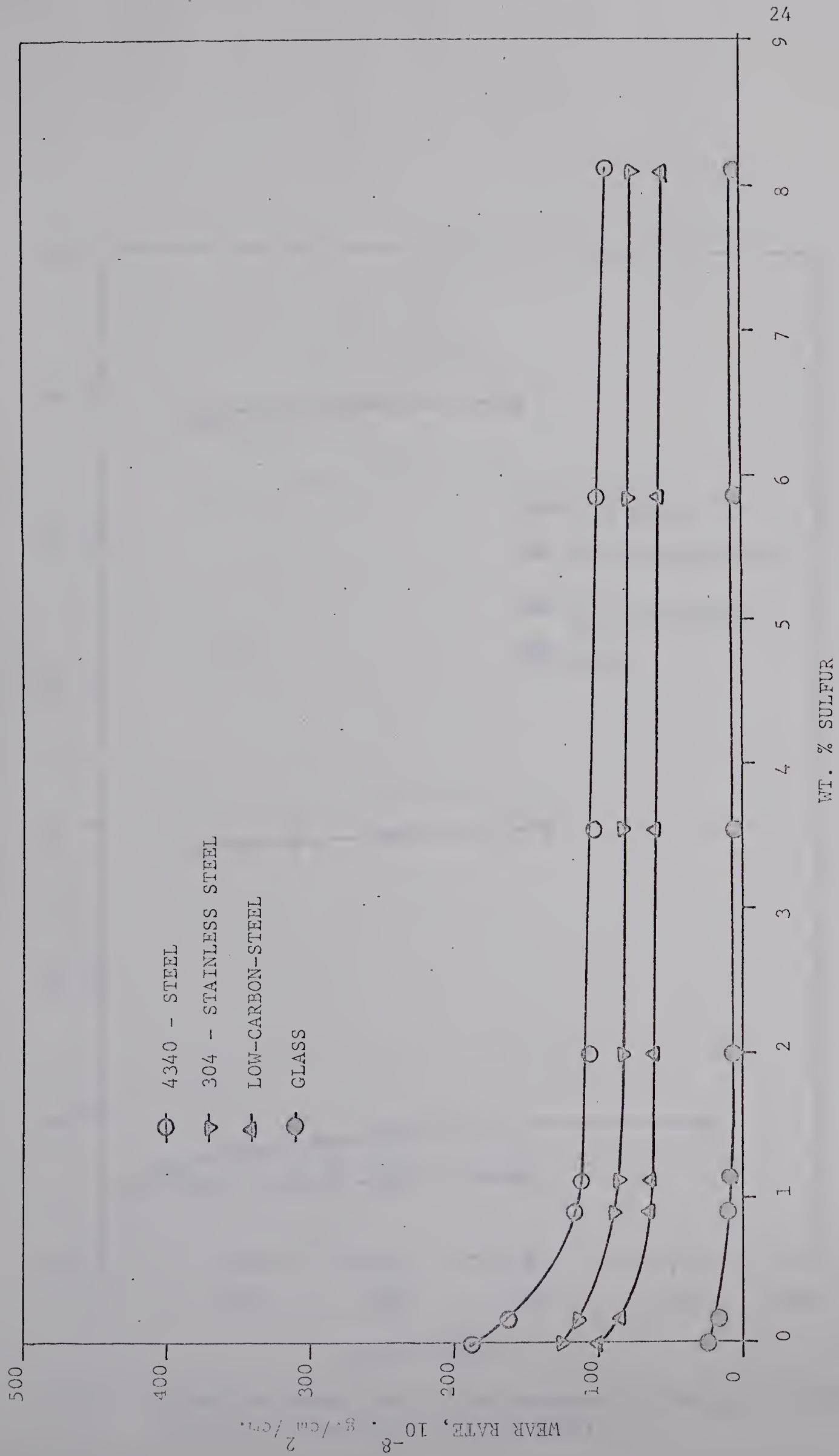


FIG. 8 EFFECT OF SULFUR CONCENTRATION ON WEAR RATE AT A SLIDING SPEED OF 240 fpm.

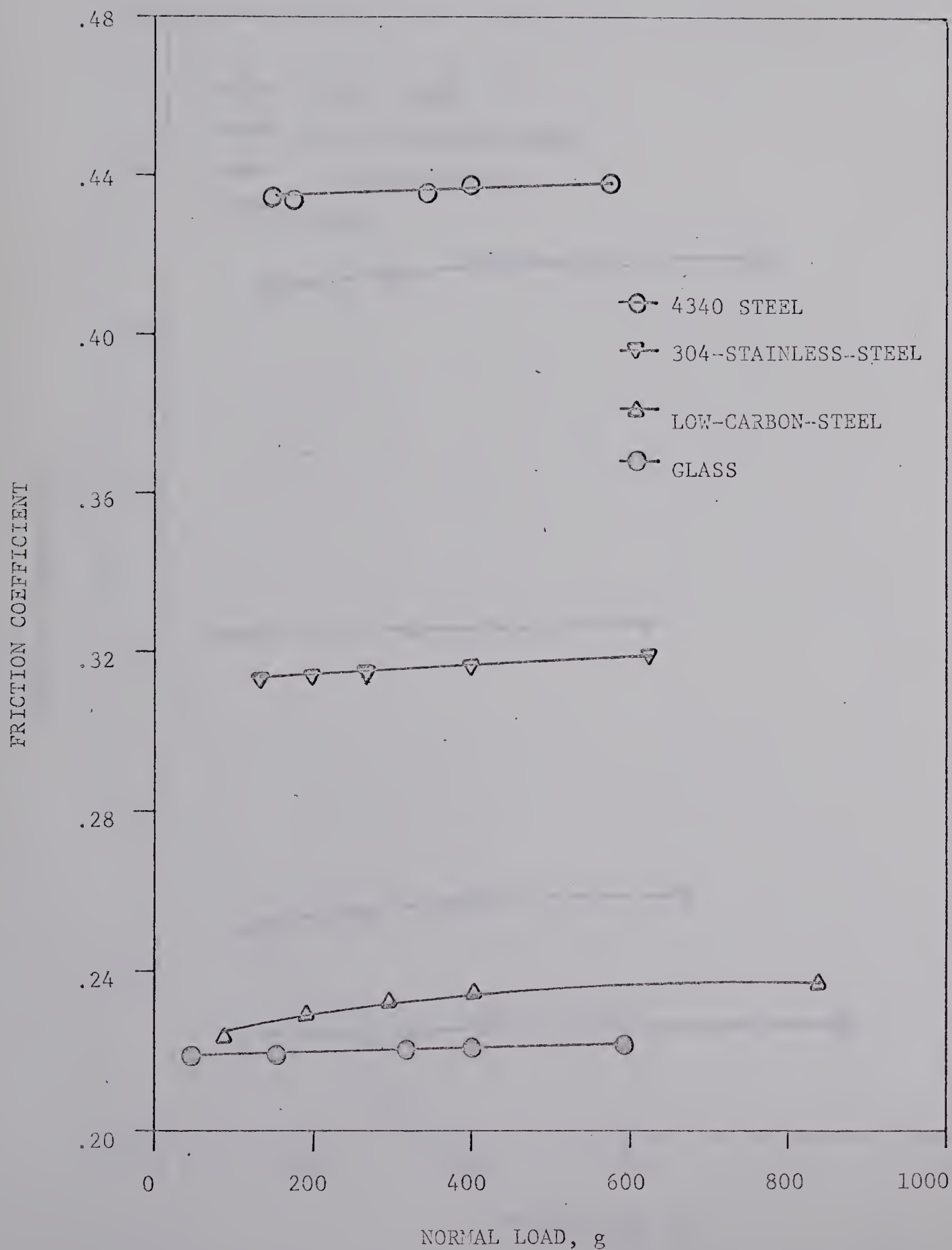


FIG. 9 - EFFECT OF NORMAL LOAD ON FRICTION COEFFICIENT AT A SLIDING SPEED OF 30 fpm FOR Fe-8.14% S ALLOY.

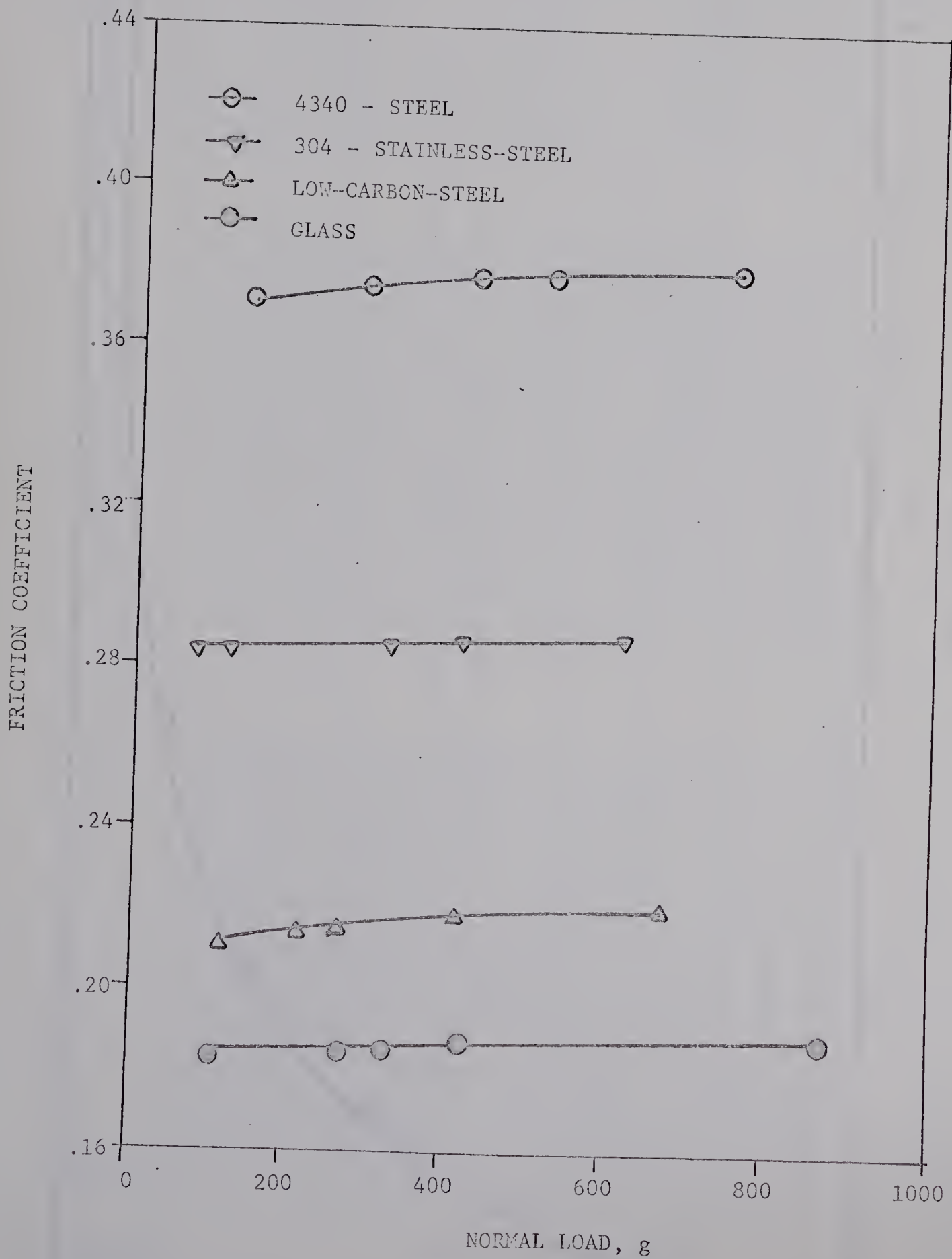


FIG. 10 - EFFECT OF NORMAL LOAD ON FRICTION COEFFICIENT AT A SLIDING SPEED OF 240 fpm FOR Fe-8.14% S ALLOY.

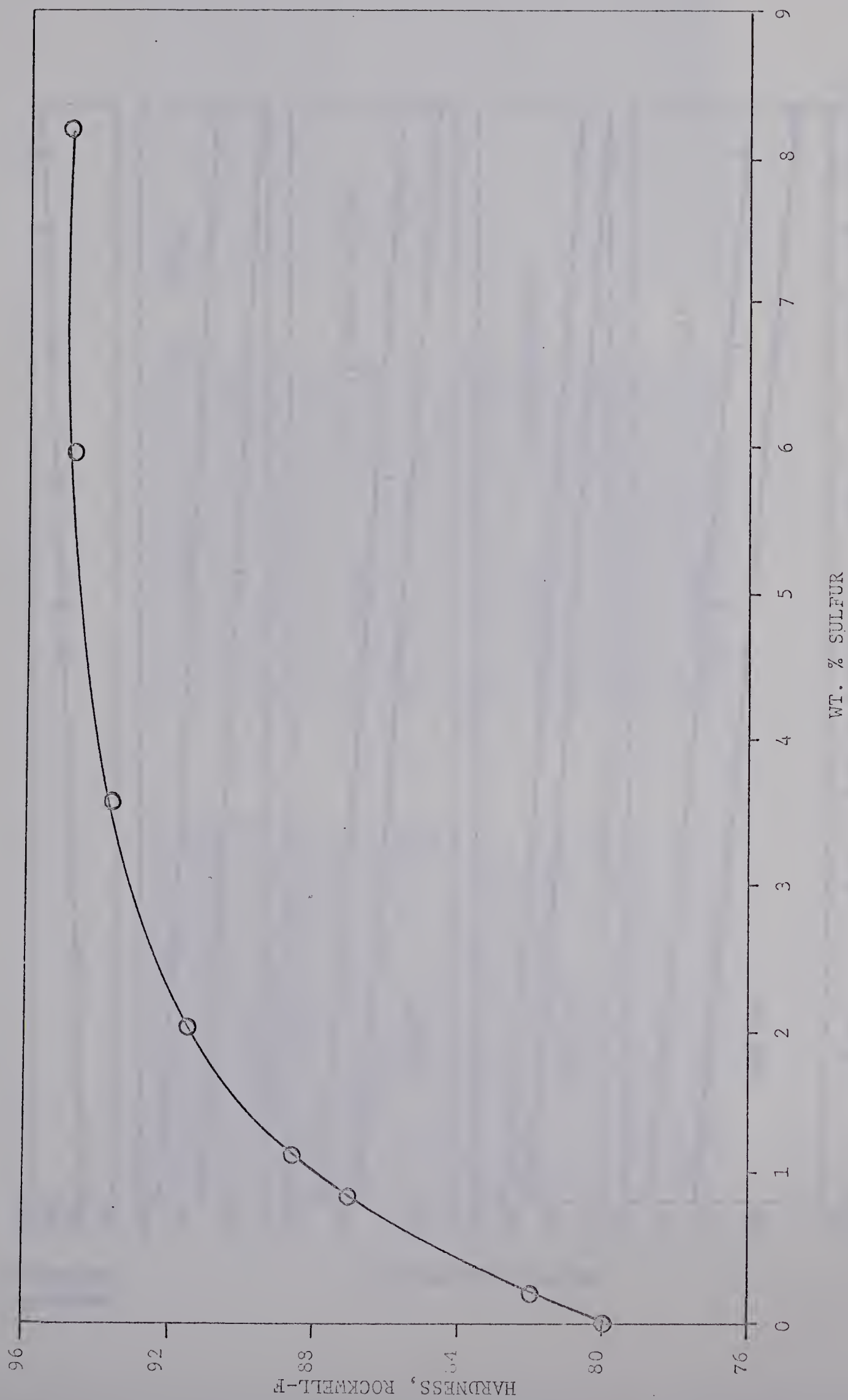


FIG. 11 - EFFECT OF SULFUR CONCENTRATION ON THE HARDNESS OF THE ALLOY

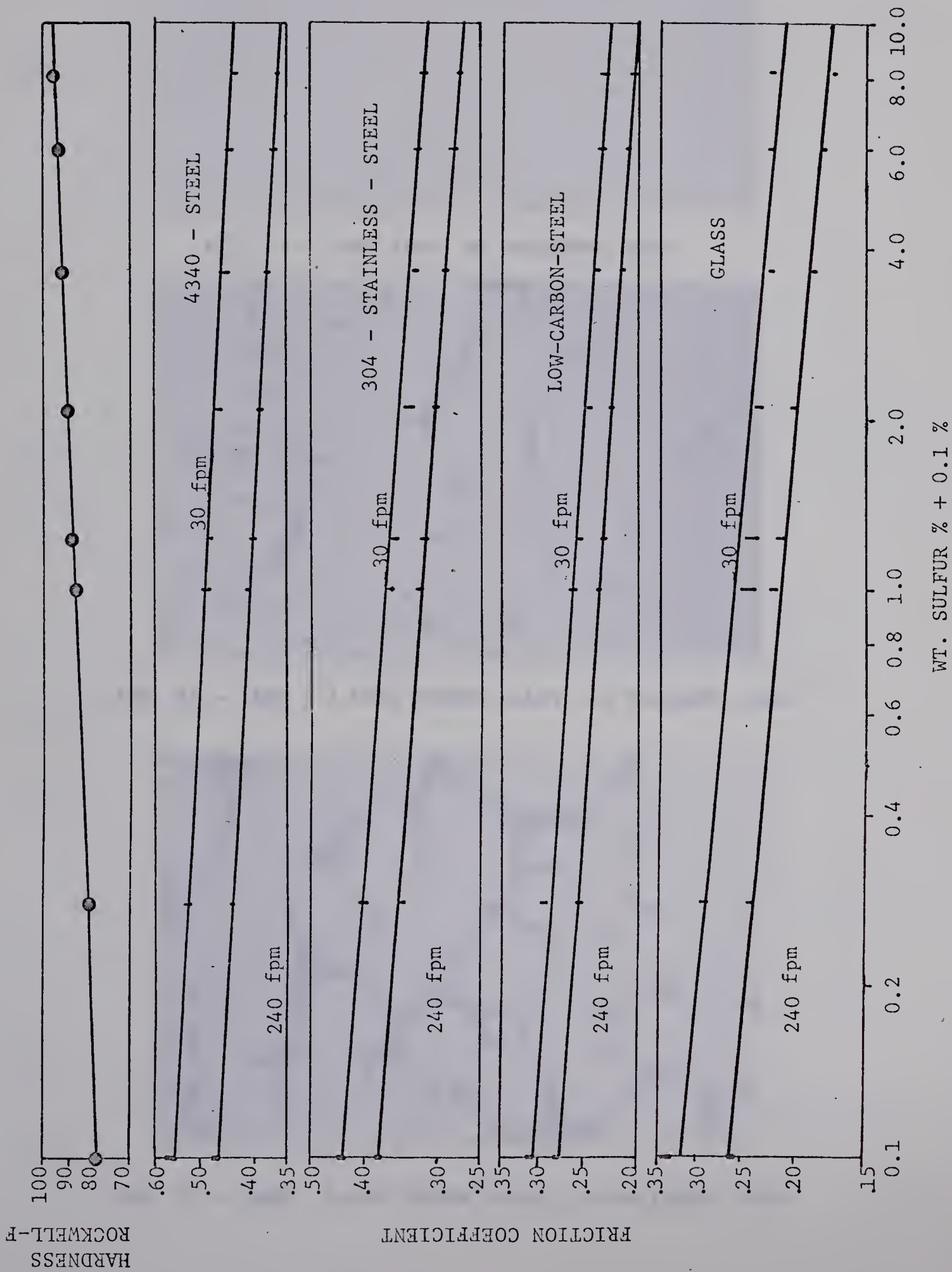


FIG. 12 - EFFECT OF SULFUR CONCENTRATION ON FRICTION COEFFICIENT AND BULK HARDNESS

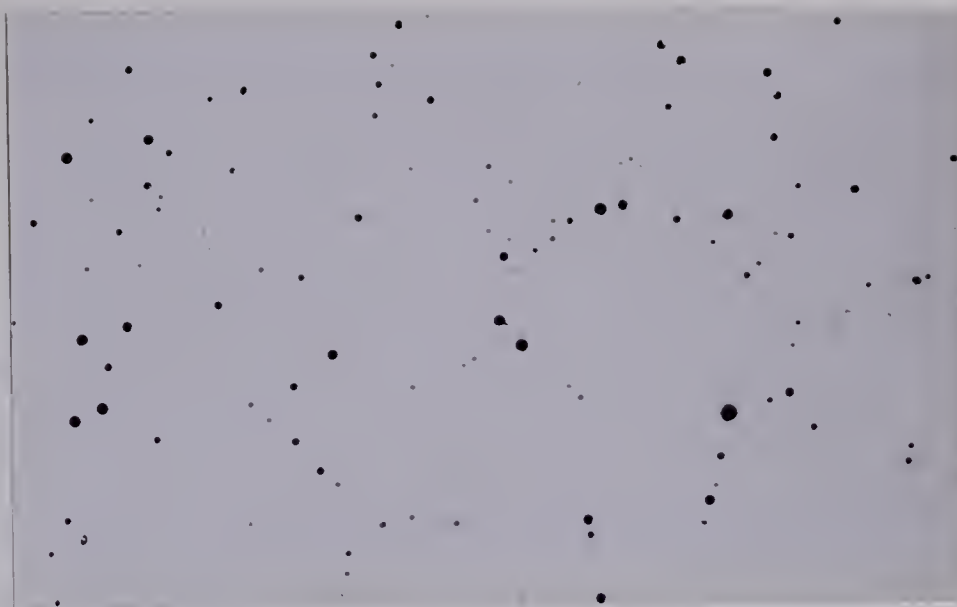


FIG. 13 - PURE IRON, AS POLISHED, X450

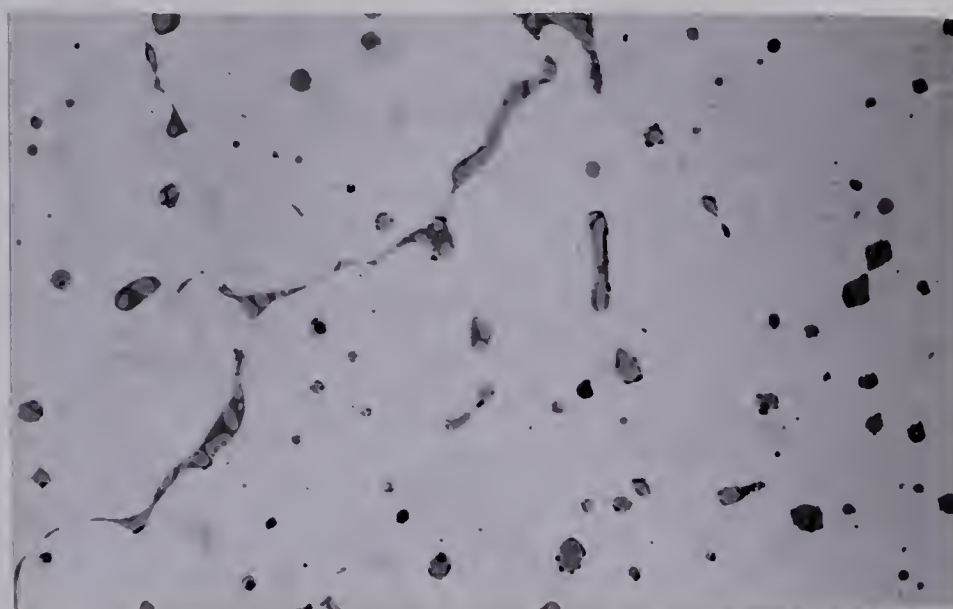


FIG. 14 - IRON - 2.005% SULFUR ALLOY, AS POLISHED, X450

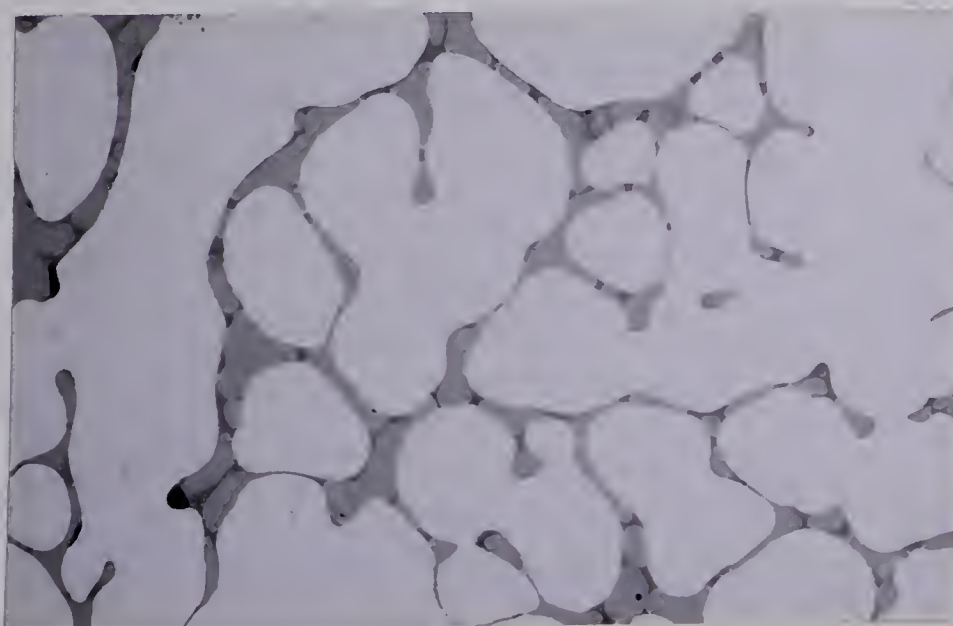


FIG. 15 - IRON - 8.14% SULFUR ALLOY, AS POLISHED, X450

DISCUSSION

The following parameters are important in friction and wear studies of two contacting solid surfaces lubricated by a solid lubricant:

1. Solid Lubricant.
2. Normal Load.
3. Friction Couple.
4. Sliding Speed.
5. Temperature.
6. Environment.

The object of the present investigation is to study the effects of the first four parameters on friction and wear at room temperature and atmospheric pressure.

Results given in figs. 3 to 11 show:

1. An apparent exponential decrease in the friction coefficient and the wear rate with increasing sulfur content in the alloy under all experimental conditions.
2. Increasing normal load increases the coefficient of friction slightly.
- 3.a. The friction coefficient and the wear rate of an alloy increase with rising hardness of the metallic-plates, forming friction couples with the specimen.
- b. The glass-specimen friction couple shows lower friction and wear than the metallic plate-specimen friction couples.
4. Increasing sliding speed reduces the friction coefficient and the wear rate.

1. The effect of solid lubricant:

The effect of sulfur concentration on friction and wear properties in the present work and in the investigation of Buckley and Johnson (1964) showed similar trends. They observed a greater decrease in friction and wear than was found in the present investigation at sulfur concentrations between 0% and 0.05%. This is a consequence of greater friction and wear of pure iron in high vacuum than in air. The increased rate of reduction of these properties with higher FeS content in the alloys in vacuum is due to the absence of surface oxide films which invariably are present and contribute to lubrication when experiments are conducted in air. The reason for the need of only 0.05% S as compared with 2.5% S in this thesis to obtain low friction and wear is the use of different friction couples in the two investigations. In their experiments both plate and rider were treated with sulfur. In this thesis, however, only the rider contained sulfur, which reduces friction and wear to a smaller extent than in the previous case (de Villemeur, 1956).

The similar trend of variation in friction and wear properties with respect to sulfur concentration in the alloy in both investigations can be explained by the adhesion theory. Increased sulfur content will increase the bulk hardness of the alloy as a result of second phase hardening, in addition, the effective shear strength and the effective adhesion energy between the two contacting surfaces will decrease as a consequence of an increase in the relative amount of FeS in the surface.

The trend of variation of friction, wear and hardness properties (Figs. 3,4,5,6,7,8 and 11) suggest an exponential relationship between these properties and the sulfur content in the alloy. This is indeed the case, as indicated by the logarithmic representation of the friction and the hardness data in Fig. 12.

The friction coefficient "f" and the bulk hardness "p_m" (Rockwell F) were expressed by the following power laws:

$$f = f_1 (S\% + 0.1\%)^{-n} \quad \text{-----} \quad \text{Eq. (5)}$$

$$p_m = p_{m,1} (S\% + 0.1\%)^q \quad \text{-----} \quad \text{Eq. (6)}$$

Where, "f₁" and "p_{m,1}" represent the friction coefficient and the bulk hardness of the alloy at 0.9% S respectively.

"n" and "q" are friction couple dependent constants and were evaluated from the slopes of the straight lines shown in Fig. 12. The arbitrary constant 0.1% in both equations were chosen to obtain the best possible fit to the experimental data and to ensure finite values of "f" and "p_m" at 0%S. Values of "p_{m,1}" and "q" were found to be 87.5 Rockwell-F hardness and 0.042 respectively. Values of "f₁" and "n" are shown in Table 12.

For the mathematical description of the dependence of the friction coefficient on the sulfur concentration, the model by Bowden, Gregory and Tabor (1945) for solid film lubrication was used, which has the following form:

$$f = a.s_m/p_m + (1 - a) .s_1/p_m \quad \text{-----} \quad \text{Eq. (7)}$$

Where, "s_m" and "s₁" are the shear strengths of the matrix and the solid lubricant respectively and "a" represents the fraction of the penetrated lubricant layer.

Since "a" could not be determined experimentally, the following equation was defined:

$$s_{\text{eff}} = a s_m + (1-a)s_1 \quad \text{-----} \quad \text{Eq. (7a)}$$

Where " s_{eff} " in kg/mm^2 is the effective shear strength of the friction couple. This permits to write Eq. 7 in the form

$$f = \frac{s_{eff}}{p_m} \text{----- Eq. (7b)}$$

Assuming that for the hardness range considered no large error is committed when the Rockwell F hardness is used in lieu of the bulk hardness, one can substitute Eq. 6 into Eq. 7b and obtains

$$f = s_{eff}/p_{m,1} (S\% + 0.1\%)^q \text{----- Eq. (8)}$$

Where " s_{eff} " represents the effective shear strength of the friction pair. It is reasonable to assume that " s_{eff} " is a decreasing function of the sulfur concentration in the alloy.

The following two expressions for " s_{eff} " can be substituted into Eq. (8)

$$s_{eff.} = s_{m,0} - \text{Constant } (\%S) \text{----- Eq. (9)}$$

$$s_{eff.} = s_{m,1} (S\% + 0.1\%)^{-r} \text{----- Eq. (10)}$$

Substitution shows that only Eq. (10) provides agreement with the experimental data because Eq. (8), then assumes the form -

$$f = s_{m,1} (S\% + 0.1\%)^{-(r+q)}/p_{m,1} \text{----- Eq. (11)}$$

Which is identical to Eq. (5) and in which "n" is the algebraic sum of the exponents "r" and "q".

This analysis shows that the decrease in friction coefficient is caused by the increase in the bulk hardness and the decrease in the shear strength of the interface with rising sulfur content in the alloys.

2. The effect of normal load:

The slight increase in the friction coefficient was due to the increase in the penetrated fraction of the lubricant layer "a" which raises the value of the effective shear strength.

3. Friction couple:

Increased hardness of the plate material resulted in higher friction and wear. This is believed to be a consequence of increased penetration of the lubricant layer by the asperities of the plate. Glass, in spite of its high hardness, showed low friction and wear characteristics because the energy of adhesion of a glass-specimen couple is always lower than that of any metal-specimen couple. From the expression for the friction coefficient and the size of the wear particle in Eqs. (2) and (4) respectively, it can be concluded that a decrease in the energy of adhesion will result in lower friction and wear.

4. The effect of sliding speed:

Friction and wear properties decreased at higher sliding speeds. This can be explained in terms of the localized temperature rise as a consequence of higher sliding speed, which lowers the shear strength of the surface film and improves the surface flow of FeS.

SUMMARY AND CONCLUSIONS

1. Addition of small amounts of sulfur to pure iron reduces the friction coefficient and the wear rate and increases the penetration hardness considerably. Rate of change of these properties with respect to the sulfur content in the alloy become lower at higher sulfur concentrations.
2. Increased sliding speed reduces the friction coefficient and the wear rate.
3. The friction coefficient has a tendency to rise slightly with increasing normal load.
4. Friction and wear of the iron-sulfur alloys are increased with rising hardness of the plate material.
5. The bulk hardness and the friction coefficient are expressed by the following power laws:

$$p_m = 87.5 (S\% + 0.1\%)^{0.042}$$

$$\text{and} \quad f = f_1 (S\% + 0.1\%)^{-n}.$$

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APPENDIX I

FORM, PURITY AND SUPPLIER OF MATERIALS:

A. IRON POWDER:	ASSAY (Fe)	-	MIN. 99.5%
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LIMITS OF IMPURITIES:

INSOLUBLE IN H ₂ SO ₄	0.20%
WATER SOLUBLE	0.03%
NITROGEN	0.003%
SULFUR	0.001%
ARSENIC	0.0005%
LEAD	0.005 %

SUPPLIER: ALLIED CHEMICAL, GENERAL CHEMICAL DIVISION, N.Y., U.S.A.

B. SULFUR FLOWER

SULFUR FLOWER CODE 2381, BAKER AND ADAMSON PRODUCT.

SUPPLIER: ALLIED CHEMICAL, GENERAL CHEMICAL DIVISION, MORRISTOWN, U.S.A.

TABLE I

EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE FOR SPECIMEN ON GLASS-PLATE AT A SLIDING SPEED OF 30 fpm

WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT	WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT
0.000	69	22	0.339	2.005	110	25	0.227
	170	58	0.341		200	46	0.230
	290	100	0.345		300	70	0.233
	402	140	0.348		400	94	0.235
	715	250	0.350		675	160	0.237
0.180	105	30	0.286	3.550	100	22	0.220
	138	40	0.290		180	40	0.222
	240	70	0.292		267	60	0.225
	410	120	0.293		395	90	0.228
	665	195	0.294		500	115	0.230
0.904	110	26	0.237	5.870	100	22	0.220
	168	40	0.238		181	40	0.221
	290	70	0.242		300	65	0.229
	408	100	0.245		400	90	0.225
	800	200	0.250		620	140	0.226
1.130	170	40	0.235	8.140	55	12	0.218
	210	50	0.238		160	35	0.219
	290	70	0.241		317	70	0.221
	410	100	0.243		407	90	0.221
	675	160	0.247		600	135	0.225

TABLE 2

EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE FOR SPECIMEN ON LOW CARBON
STEEL-PLATE AT A SLIDING SPEED OF 30 fpm

WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT	WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT
0.000	115	40	0.348	2.005	145	35	0.242
	170	60	0.352		205	50	0.244
	284	100	0.353		325	80	0.246
	410	145	0.354		405	100	0.248
	565	200	0.354		565	145	0.248
0.180	240	75	0.312	3.550	107	25	0.234
	300	95	0.317		200	47	0.235
	330	105	0.318		300	71	0.237
	400	130	0.326		400	96	0.240
	600	195	0.326		700	170	0.243
0.904	118	35	0.255	5.870	130	30	0.231
	195	50	0.257		250	58	0.232
	250	65	0.260		300	70	0.233
	330	100	0.264		400	95	0.235
	755	200	0.265		700	165	0.236
1.130	82	20	0.296	8.140	130	29	0.223
	200	50	0.250		193	45	0.229
	315	80	0.254		300	70	0.233
	392	100	0.255		405	95	0.235
	590	150	0.255		850	200	0.236

TABLE 3

EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE FOR SPECIMEN ON 304-STAINLESS STEEL-PLATE
AT A SLIDING SPEED OF 30 fpm.

WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT	WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT
0.000	70	30	0.430	2.005	180	60	0.333
	150	65	0.434		208	70	0.336
	250	110	0.440		250	85	0.340
	395	175	0.444		410	140	0.342
	675	300	0.444		645	220	0.342
0.180	126	50	0.397	3.550	126	40	0.325
	200	80	0.400		290	95	0.328
	300	120	0.401		350	115	0.329
	420	170	0.404		400	132	0.330
	740	300	0.406		600	200	0.334
0.904	90	32	0.356	5.870	123	40	0.325
	195	70	0.359		215	70	0.326
	220	80	0.364		275	90	0.328
	398	145	0.364		410	135	0.330
	550	200	0.364		605	200	0.331
1.130	115	40	0.348	8.140	65	20	0.312
	200	70	0.350		207	65	0.319
	310	110	0.355		270	85	0.315
	405	145	0.358		395	125	0.317
	615	220	0.358		625	200	0.320

TABLE 4

EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE FOR SPECIMEN ON 4340-STEEL-PLATE
AT A SLIDING SPEED OF 30 fpm

WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT	WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT
0.000	100	55	0.550	2.005	55	25	0.458
	172	95	0.551		210	97	0.462
	290	160	0.553		350	162	0.464
	415	230	0.554		408	190	0.465
	630	350	0.556		600	208	0.467
0.180	120	63	0.522	3.550	190	85	0.447
	200	105	0.525		250	112	0.448
	350	185	0.529		400	180	0.450
	415	220	0.530		500	226	0.452
	660	350	0.531		750	340	0.454
0.904	72	35	0.483	5.870	72	32	0.439
	175	85	0.485		216	95	0.440
	350	170	0.486		396	175	0.442
	410	200	0.488		520	230	0.442
	610	300	0.492		700	310	0.444
1.130	120	57	0.475	8.140	150	65	0.433
	210	100	0.476		173	75	0.434
	334	160	0.479		345	150	0.435
	400	192	0.480		400	175	0.438
	600	290	0.484		570	250	0.439

TABLE 5

EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE FOR SPECIMEN ON GLASS-PLATE
AT A SLIDING SPEED OF 240 fpm

WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT	WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT
0.000	95	24	0.253	2.005	75	15	0.200
	235	60	0.255		197	40	0.203
	350	90	0.257		250	51	0.204
	407	105	0.258		392	80	0.204
	850	220	0.259		635	130	0.205
0.180	84	20	0.238	3.550	54	10	0.185
	200	48	0.240		160	30	0.188
	310	75	0.242		360	68	0.189
	390	95	0.244		400	76	0.190
	695	170	0.245		550	105	0.191
0.904	92	20	0.218	5.870	85	15	0.177
	200	44	0.220		226	40	0.177
	270	60	0.222		308	55	0.179
	400	90	0.225		390	70	0.179
	600	135	0.225		770	140	0.182
1.130	95	20	0.211	8.140	88	15	0.171
	210	45	0.214		260	45	0.174
	350	75	0.214		310	54	0.174
	395	85	0.215		400	70	0.175
	600	130	0.217		850	150	0.177

TABLE 6

EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE FOR SPECIMEN ON LOW CARBON
STEEL-PLATE AT A SLIDING SPEED OF 240 fpm

WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT	WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT
0.000	56	15	0.268	2.005	94	20	0.213
	185	50	0.270		140	30	0.214
	350	90	0.273		300	65	0.217
	404	110	0.273		390	85	0.218
	710	195	0.275		550	120	0.219
0.180	80	20	0.250	3.550	96	20	0.208
	150	38	0.253		190	40	0.210
	350	90	0.257		330	70	0.212
	407	105	0.258		400	85	0.212
	770	200	0.260		750	160	0.214
0.904	100	33	0.230	5.870	98	20	0.204
	150	35	0.234		145	30	0.207
	340	80	0.235		215	45	0.209
	403	95	0.236		405	85	0.210
	840	200	0.238		715	150	0.210
1.130	67	15	0.224	8.140	100	20	0.200
	220	50	0.227		197	40	0.203
	350	80	0.229		245	50	0.204
	392	90	0.229		400	85	0.207
	800	185	0.232		650	135	0.208

TABLE 7

EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE FOR SPECIMEN ON 304-STAINLESS
STEEL-PLATE AT A SLIDING SPEED OF 240 fpm

WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT	WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT
0.000	85	30	0.853	2.005	85	25	0.294
	225	80	0.355		135	40	0.296
	310	110	0.356		300	90	0.300
	420	150	0.358		415	125	0.301
	750	270	0.360		760	230	0.303
0.180	74	25	0.338	3.550	70	20	0.286
	190	65	0.342		209	60	0.287
	350	120	0.343		330	95	0.288
	407	140	0.344		415	120	0.290
	610	210	0.345		550	160	0.291
0.904	50	16	0.320	5.870	55	15	0.274
	170	55	0.323		200	55	0.275
	340	110	0.323		290	80	0.276
	400	130	0.325		395	110	0.279
	600	195	0.325		710	200	0.282
1.130	80	25	0.313	8.140	63	17	0.270
	175	55	0.314		110	30	0.273
	270	85	0.315		310	85	0.274
	397	125	0.315		400	110	0.275
	760	240	0.316		600	165	0.275

TABLE 8

EFFECT OF SULFUR CONCENTRATION AND NORMAL FORCE FOR SPECIMEN ON 4340-STEEL-PLATE
AT A SLIDING SPEED OF 240 fpm

WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT	WEIGHT % SULFUR	NORMAL LOAD, g	FRICTION FORCE, g	FRICTION COEFFICIENT
0.000	100	45	0.450	2.005	130	50	0.385
	210	95	0.452		180	70	0.389
	320	145	0.454		270	105	0.389
	395	180	0.456		410	160	0.391
	700	320	0.457		690	270	0.392
0.180	150	65	0.434	3.550	120	45	0.375
	230	100	0.435		265	100	0.377
	275	120	0.437		290	110	0.380
	395	175	0.438		420	160	0.382
	750	330	0.440		550	210	0.382
0.904	135	55	0.407	5.870	150	55	0.367
	195	80	0.410		190	70	0.368
	280	115	0.412		270	100	0.370
	400	165	0.414		405	150	0.371
	750	310	0.414		700	260	0.372
1.130	100	40	0.400	8.140	125	45	0.360
	210	85	0.405		275	100	0.364
	320	130	0.407		410	150	0.366
	405	165	0.408		505	190	0.367
	735	300	0.409		735	270	0.368

TABLE 9

EFFECT OF SULFUR CONCENTRATION ON WEAR RATE OF Fe-S ALLOYS AT A SLIDING SPEED OF 30 fpm

GLASS		LOW-CARBON-STEEL				304-STAINLESS-STEEL				4340-STEEL				
WEIGHT % SULFUR	WEIGHT LOSS, g X 10 ⁻⁸	WEAR RATE g/cm ² /cm X 10 ⁻⁸	WEIGHT % SULFUR	WEIGHT LOSS, g X 10 ⁻⁸	WEAR RATE g/cm ² /cm X 10 ⁻⁸	WEIGHT % SULFUR	WEIGHT LOSS, g X 10 ⁻⁸	WEAR RATE g/cm ² /cm X 10 ⁻⁸	WEIGHT % SULFUR	WEIGHT LOSS, g X 10 ⁻⁸	WEAR RATE g/cm ² /cm X 10 ⁻⁸	WEIGHT % SULFUR	WEIGHT LOSS, g X 10 ⁻⁸	WEAR RATE g/cm ² /cm X 10 ⁻⁸
0.000	0.0030	24.7	0.000	0.0176	145	0.000	0.0250	206	0.000	0.0500	412			
0.180	0.0020	16.5	0.180	0.0156	128	0.180	0.0200	169	0.180	0.0417	343			
0.904	0.0013	10.7	0.904	0.0125	103	0.904	0.0147	121	0.904	0.0275	226			
1.130	0.0012	9.9	1.130	0.0114	94	1.130	0.0142	117	1.130	0.0260	214			
2.005	0.0010	8.2	2.005	0.0100	82	2.005	0.0136	112	2.005	0.0235	193			
3.550	0.0009	7.4	3.550	0.0093	76	3.550	0.0128	105	3.550	0.0205	168			
5.870	0.0008	6.6	5.870	0.0084	69	5.870	0.0119	98	5.870	0.0185	151			
8.140	0.0007	5.8	8.140	0.0077	63	8.140	0.0110	91	8.140	0.0175	144			

* TIME OF WEAR = 1/2 HOUR

TABLE 10

EFFECT OF SULFUR CONCENTRATION ON WEAR RATE OF Fe-S ALLOYS AT A SLIDING SPEED OF 240 fpm

GLASS		LOW-CARBON-STEEL				304-STAINLESS-STEEL				4340-STEEL			
* WEIGHT % SULFUR LOSS,g	WEAR RATE $\text{g/cm}^2/\text{cm}$ $\times 10^{-8}$	WEIGHT % SULFUR LOSS,g	WEAR RATE $\text{g/cm}^2/\text{cm}$ $\times 10^{-8}$	WEIGHT % SULFUR LOSS,g	WEAR RATE $\text{g/cm}^2/\text{cm}$ $\times 10^{-8}$	WEIGHT % SULFUR LOSS,g	WEAR RATE $\text{g/cm}^2/\text{cm}$ $\times 10^{-8}$	WEIGHT % SULFUR LOSS,g	WEAR RATE $\text{g/cm}^2/\text{cm}$ $\times 10^{-8}$	WEIGHT % SULFUR LOSS,g	WEAR RATE $\text{g/cm}^2/\text{cm}$ $\times 10^{-8}$	WEIGHT % SULFUR LOSS,g	WEAR RATE $\text{g/cm}^2/\text{cm}$ $\times 10^{-8}$
0.000	0.0200	20.6	0.000	0.1000	103	0.000	0.1300	134	0.000	0.1800	185		
0.180	0.0120	12.4	0.180	0.0855	85	0.180	0.1068	110	0.180	0.1550	160		
0.904	0.0086	8.9	0.904	0.0610	63	0.904	0.0860	89	0.904	0.1112	115		
1.130	0.0080	8.2	1.130	0.0595	61	1.130	0.0820	85	1.130	0.1070	110		
2.005	0.0070	7.2	2.005	0.0580	60	2.005	0.0785	81	2.005	0.1000	103		
3.550	0.0065	6.7	3.550	0.0563	58	3.550	0.0760	78	3.550	0.0970	100		
5.870	0.0062	6.4	5.870	0.0550	57	5.870	0.0750	77	5.870	0.0945	97		
8.140	0.0058	6.0	8.140	0.0530	55	8.140	0.0730	75	8.140	0.0930	96		

* TIME OF WEAR = 1/2 HOUR

TABLE 11

EFFECT OF SULFUR CONCENTRATION ON BULK HARDNESS OF Fe-S ALLOYS

WEIGHT % SULFUR	0.000	0.180	0.904	1.130	2.005	3.550	5.870	8.140
BULK HARDNESS ROCKWELL "F"	80.0	82.0	86.7	88.8	91.8	94.0	95.0	95.5

TABLE 12

VALUES OF "f₁", AND "n" UNDER DIFFERENT SLIDING CONDITIONS

PLATE MATERIAL	GLASS		LOW-CARBON-STEEL		304-STAINLESS STEEL		4340-STEEL	
SLIDING SPEED	30 fpm	240 fpm	30 fpm	240 fpm	30 fpm	240 fpm	30 fpm	240 fpm
f ₁	0.258	0.218	0.260	0.233	0.365	0.316	0.485	0.405
n	0.0878	0.0878	0.0683	0.0683	0.0725	0.0725	0.0551	0.0551

* Values of 'f₁', and 'n' are obtained from fig. 12 and their physical meaning is given in Eq. (5).

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